Transport Roads & Maritime Services

NSW GOVERNMENT

Test method T1017

Determination of metallic zinc

NOVEMBER 2012

Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary Added	D. Dash	Jun 2001
Ed 2/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T1017 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T1017

Determination of metallic zinc

1. Scope

This test method sets out the procedure for determination of metallic zinc by the generation of hydrogen. The test method is identical with the method outlined in Appendix B of the British Standard 3982-1966. It is applicable to Zinc dust and Zinc rich pigment recovered from Zinc rich paints.

2. Apparatus

(a) A gas measuring apparatus consisting of a water-jacketed gas burette of 400 mL capacity graduated from 300 mL.

(b) A water-cooled reaction flask of 400 mL capacity. Any suitable apparatus may be used but that illustrated in B.S. 3982 is a convenient type.

(c) A boat of a suitable plastic material such as polyethylene.

(d) Agate mortar and pestle.

(e) 75 μm AS sieve.

3. Reagents

The reagents used are to be of a recognised analytical reagent quality.

(a) Distilled water, or water otherwise prepared of equal quality.

(b) Hydrochloric acid/cuprous chloride reagent. To prepare dissolve 0.05 g of cuprous chloride (Cu_2Cl_2) in 700 mL of hydrochloric acid (d = 1.18 g/mL) and dilute to 1000 mL with water.

CAUTION: Always add acid to the water, slowly and carefully - NEVER THE REVERSE. Safety glasses must be worn.

4. Levelling Solution

Acidify some water with a few drops of hydrochloric acid and add sufficient methyl orange solution to render the colour of the water in the apparatus readily visible.

5. Procedure

(a) Pass a steady flow of tap water through the water jackets of the burette.
Procedure

(b) Introduce 30 mL of hydrochloric acid-cuprous chloride reagent into the reaction flask and clamp it in a horizontal position.

(c) If the sample has been collected by scraping fresh paint off the surface, and placed in a tightly sealed container, it may contain ethanol which will affect the result. In such cases dry the sample at room temperature and 60% relative humidity for 24 hours.

Coarse material requires a longer reaction time. In such cases grind the material to pass a 75 μm sieve using an agate mortar and pestle. The sample must be free from solvent and properly cured before testing.

(d) Weigh to the nearest milligram approximately 1 g of the sample in the plastic boat and introduce into the neck of the reaction flask so that it floats on the acid solution taking care that the sample does not come into contact with the solution.

(e) Replace the stopper and connection from the flask to the measuring tube.

(f) When constant temperature has been established by the circulation of tap water adjust the pressure in the reaction flask to atmospheric by opening the stopcock connecting the flask and measuring tube and lowering the levelling bottle until the levels are the same in the measuring tube and bottle.

(g) Adjust the stopcock to connect the measuring tube to the atmosphere and raise the levelling bottle to expel all air from the measuring tube.

(h) Turn the cock to connect the measuring tube and the reaction flask once more and place the levelling bottle in the rest position above the measuring tube.

(i) Tilt the reaction flask to drop the boat and sample into the acid solution. Shake the flask well.

(j) When the reaction appears to be complete, again shake the flask well and check that the level in the measuring tube remains constant.

(k) Lower the levelling bottle until the levels in the bottle and measuring tube are equal.

(l) Record the volume of hydrogen, the barometric pressure and the temperature of the cooling water. From the temperature of the cooling water read off the saturated water vapour pressure at that temperature from Table A.

6. Calculation and Report

Calculate the percentage of metallic zinc (Zn) as follows:

$$\text{Metallic Zinc (Zn) per cent by mass} = \frac{0.1048V(P-S)}{M(T+273)}$$

Where:

V = Volume in mL of hydrogen liberated.

P = Barometric pressure in mm of Mercury.

T = Temperature of cooling water °C.

S = Saturated water vapour pressure in mm of Mercury at T°C.

M = Mass in g of sample.

Report the percentage by mass of metallic zinc (Zn) present as the mean of the replicate determination which should not vary by more than 0.5%.

SATURATED WATER VAPOUR PRESSURES, IN MILLIMETRES OF MERCURY

Temperature °C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	4.6	4.6	4.6	4.7	4.7	4.7	4.8	4.8	4.9	4.9
1	4.9	5.0	5.0	5.0	5.1	5.1	5.1	5.2	5.2	5.3
2	5.3	5.3	5.4	5.4	5.4	5.5	5.5	5.6	5.6	5.6
3	5.7	5.7	5.8	5.8	5.8	5.9	5.9	6.0	6.0	6.0
4	6.1	6.1	6.2	6.2	6.3	6.3	6.4	6.4	6.5	6.5
5	6.5	6.6	6.6	6.7	6.7	6.8	6.8	6.9	6.9	7.0
6	7.0	7.1	7.1	7.2	7.2	7.3	7.3	7.4	7.4	7.5
7	7.5	7.6	7.6	7.7	7.8	7.8	7.9	7.9	7.9	8.0
8	8.0	8.1	8.2	8.2	8.3	8.3	8.4	8.4	8.5	8.6
9	8.6	8.7	8.7	8.8	8.8	8.9	9.0	9.0	9.1	9.1
10	9.2	9.3	9.3	9.4	9.5	9.5	9.6	9.7	9.7	9.8
11	9.8	9.9	10.0	10.0	10.1	10.2	10.2	10.3	10.4	10.4
12	10.5	10.6	10.7	10.7	10.8	10.9	10.9	11.0	11.1	11.2
13	11.2	11.3	11.4	11.5	11.5	11.6	11.7	11.8	11.8	11.9
14	12.0	12.1	12.1	12.2	12.3	12.4	12.5	12.5	12.6	12.7
15	12.8	12.9	13.0	13.0	13.1	13.2	13.3	13.4	13.5	13.5
16	13.6	13.7	13.8	13.9	14.0	14.1	14.2	14.3	14.3	14.4
17	14.5	14.6	14.7	14.8	14.9	15.0	15.1	15.2	15.3	15.4
18	15.5	15.6	15.7	15.8	15.9	16.0	16.1	16.2	16.3	16.4
19	16.5	16.6	16.7	16.8	16.9	17.0	17.1	17.2	17.3	17.4
20	17.5	17.6	17.7	17.9	18.0	18.1	18.2	18.3	18.4	18.5
21	18.7	18.8	18.9	19.0	19.1	19.2	19.3	19.5	19.6	19.7
22	19.8	19.9	20.1	20.2	20.3	20.4	20.6	20.7	20.8	20.9
23	21.1	21.2	21.3	21.5	21.6	21.7	21.8	22.0	22.1	22.2
24	22.4	22.5	22.7	22.8	22.9	23.1	23.2	23.3	23.5	23.6